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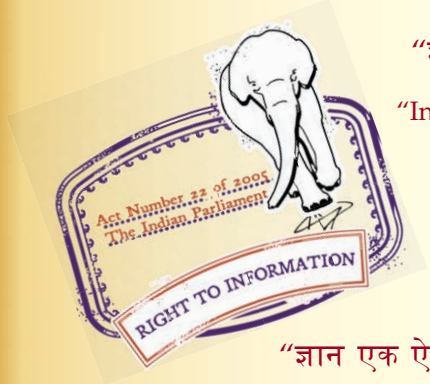
IS 8025 (1990): Monocrotophos, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



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IS 8025 : 1990
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भारतीय मानक

मोनोक्रोटोफोस, तकनीकी – विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

MONOCROTOPHOS, TECHNICAL — SPECIFICATION

(*Second Revision*)

First Reprint FEBRUARY 1992

UDC 632'951 MON

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN. 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 1991

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 25 May 1990, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

Monocrotophos, technical, exists in two isomeric forms, namely, *cis* isomer and *trans* isomer. Of these two isomeric forms, only the *cis* isomer is effective. This is a systemic as well a$ contact insecticide and employed in the preparation of pesticidal formulations for the control of insect and acarine pests of agricultural crops.

This standard, published in 1976, was first revised in 1983. This revision has been taken up to update the various requirements in the light of the experience gained.

Monocrotophos is the name accepted by the International Organization for Standardization (IS0) for the insecticidal chemical dimethyl phosphate ester with (E)-3-hydroxy-N-methylcrotonamide or 3-hydroxy-N-methylcrotonamide dimethyl phosphate or dimethyl *cis*-1-methyl-2-(methyl-carbamoyl) vinyl phosphate or 3-(dimethoxy-phosphinyloxy)-N-methylisocrotonamide. The empirical and structural formulae and molecular mass of monocrotophos are as indicated below:

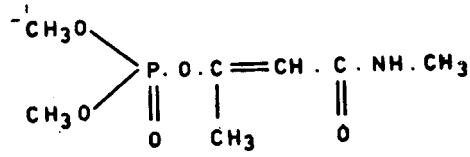
Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_7H_{14}NO_5P$		223'17

Fig. 1

In the preparation of this standard, due consideration has been given to the provisions of the ***Insecticides Act, I968*** and Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (***revised***)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MONOCROTOPHOS, TECHNICAL — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This Indian Standard prescribes the requirements and the methods of sampling and test for monocrotophos, technical.

2 REFERENCES

2.1 The Indian Standards listed in Annex A are necessary adjuncts to this standard.

3 REQUIREMENTS

3.1 Description

3.1.1 The material shall be in the form of brown viscous liquid or crystalline semi-solid.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Mooocrotopbos, Technical

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Clause No. of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	Monocrotophos content, percent by mass, *Min*	68.0	B	—
ii)	Free-acetic acid monomethylamide (MMA), percent by mass, *Max*	4·0	c	—
iii)	Acidity (H_2SO_4), percent by mass, *Max*	3·0	—	13.5.4

4 SAMPLING

4.1 Representative samples of the msterial shall be drawn in accordance with IS 10946 : 1984.

5 TESTS

5.1 Tests shall be carried out by the methods referred in col 4 and 5 of Table 1.

5.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

6 PACKING

6.1 The material shall be packed according to the requirements given in IS 8190 (Part 2) : 1988.

7 MARKING

7.1 The containers shall be securely closed and shall bear legibly and indelibly the following information in addition to the information as required under tlie ***Insecticides Act, 1968*** and Rules:

a) Name of the material;

b) Indication of the source of manufacture;

c) Date of manufacture 'and date of expiry;

d) Batch number;

e) Net volume of contents;

f) Monocrotophos content, percent (***m/m***); and

g) The minimum cautionary notice as worded in ***Insecticides Act, 1968*** and Rules.

ANNEX A

(Clause 2.1)

IS No.	*Title*	*IS No.*	*Title*
1070 : 1977	Water for general laboratory use (***second revision***)	8190 (Part 2): 1988	Requirements for packing of pesticides : Part 2 Liquid pesticides (***second revision***)
6940 : 1982	Method of tests for pesticides and their formulations (***first revision***)	10946 : 1984	Method of sampling for technical grade pesticides

ANNEX B

[*Table* 1, *Item* (i)]

DETERMINATION OF MONOCROTOPHOS CONTENT

B-l PRINCIPLE

Monocrotophos is hydrolyzed in an alkaline medium, and as a result, aceto-acetic acid monomethylamide (MMA) forms, which is quantitatively determined. colorimetrically as Iron (III) complex. Free MMA is determined separately and subtracted.

B-2 REAGENTS

B-2.1 Methanol

B-2.2 Standard Sodium Hydroxide Solution, 5 N.

B-2.3 Nitric Acid, 1 N.

B-2.4 Glacial Acetic Acid

B-2.5 Acetic Acid in Methanol Solution, 5 percent (v/v) prepared by diluting IO ml of glacial acetic acid with methanol to a volume of 200 ml.

B-2.6 **Reference Standard of Aceto-Acetic Acid Monomethylamide (MMA),** of purity not less than 98 percent.

NOTE — Anhydrous MMA standard samples are susceptible to degradation by ingress of moisture and air during storage. Store in a cool, dry and dark place. Avoid repeated opening of bottle containing the reference MMA:

B-2.6.1 ***Preparation of Pure MMA from 50 Percent Aqueous*** Solution

B-2.6.1.1 Take 200 g of the material (50 percent MMA) in the flask and attach it to vacuum rotatory pump. Evaporate water from the flask maintaining temperature of the bath at 60°C, till the moisture is 1 to 2 percent only. This can be verified on weighing the receiver flask, which should contain at least 98 g of the distilled off material. Take out the flask containing the material and dissolve it in minimum amount of warm benzene. Filter this solution through Buchner funnel and collect the filtrate. Adjust the volume of benzene in the filtrate to three times the mass of the material and leave it overnight in the freezer. Next day the crystals appear in the solution which are filtered through Buchner funnel and the filtrate is kept in the freezer for further crystallisation.

B-2.6.1.2 Two crops of crystals are obtained from the mother liquor. The crystals are pooled and kept overnight in vacuum at room temperature to dry. On the following day determine the purity by taking 0'2 g of the material, adding to it 100 ml of reagent mixture (10 parts of acetic acid and 1 part of hydrochloric acid) and 10 ml of saturated concentrated solution of potassium chloride. Titrate potentiometrically standard sodium nitrate solution at polarisation current of 5 μA and potential drop of 650 mV.

1 ml of 0'1 N $NaNO_2$ = 11'51 mg of MMA

B-2.7 **Ferric Chloride Solution**

Prepared by dissolving 40'5 g of ferric chloride ($FeCl_3 \cdot 6H_2O$) in methanol and made up to 1 litre.

B-2.8 **Phenolphthalein Indicator Solution**

Prepared by dissolving 0'2 g of phenolphthalein in 95 percent ethanol and made up to **100** ml.

B-2.9 Standard Solutiou of MMA or Monocrotophos

Weigh about 0·15 g accurately of reference standard MMA and dissolve in methanol in a 250-ml volumetric flask and make up the volume.

B-2.10 Sodium Nitrate Solution

Measure IO ml of 5 N sodium hydroxide into a 250-ml volumetric flask, 50 ml of water, 1 ml of the indicator solution, neutralize with 1 N nitric acid and make up to the mark with water.

B-2.11 Blank Solution

In a 100-ml volumetric flask mix 10 ml of sodium nitrate solution with 10 ml of acetic acid in methanol and IO ml of ferric chloride solution and make up to the mark with methanol.

B-2.12 *Standard Sodium Nitrate Solution, 0'1 N.

B-2.13 **Hydrochloric** Acid, concentrated.

B-3 PROCEDURE

B-3.1 Weigh accurately a quantity of the sample equivalent to about 1'5 g of monocrotophos as active ingredient, transfer into a 100-ml volumetric flask and make up to the mark with methanol. This stock solution is also used for free MMA determination (see Annex C). Pipette 10 ml of this dilution into a 250-ml volumetric flask, and 10 ml standard solution hydroxide solution, shake thoroughly and allow to stand at 25 ± 5°C for 30 minutes. Then add 1'0 ml of phenolphthalein indicator and titrate with 1 N nitric acid until the violet colour disappears. Temperature. during neutralization should be maintained at 25°C. Then fill the volumetric flask to the mark with water. Pipette 10 ml of this solution into a 100-ml volumetric flask and successive 10 ml of acetic acid in

methanol solution, 50 ml methanol and 10 ml of the ferric chloride solution. Make up the volume with methanol and mix well. This constitutes the test solution.

B-3.1.1 Pour 10 ml of the standard solution of MMA into a IOO-ml volumetric flask. Add IO ml of sodium nitrate solution, 10 ml acetic acid in methanol solution, 50 ml of methanol and 10 ml of the ferric chloride solution. Fill with methanol to the mark. This constitutes the comparison solution. Allow the sample solution as well as the comparison solution to stand at 25 ± 5°C for 10 minutes and measure their absorbances in a spectrophotometer at 544 nm wavelength and a l-cm cell.

NOTE — The test solution and the comparison solution should be prepared at the same time. The measurement of the absorbances of both sclutions has to be carried cut successively at short intervals. From the preparatien of the solutions to the measurement, not more than 30 minutes should elapse.

B-4 CALCULATION

B-4.1 Monocrotophos content, percent by mass

$$= 1940 \times \frac{At \times ms \times P}{As \times mt \times 100} - K$$

where

At = absorbance of test solution;

ms = mass, in g, of the reference standard MMA (100 percent purity basis);

As = absorbance of the standard reference comparison solution of MMA;

mt = mass, in g, of the sample taken for the test;

P = purity of the reference standard MMA; and

K = correction value of monocrotophos content due to free MMA (see Annex C).

ANNEX C

[*Table* 1, Item (ii) and B-3.1]

DETERMINATION OF FREE ACETO-ACETIC ACID MONOMETHYL AMIDE (MMA)

C-I REAGENTS

C-l.1 Methanol

C-l.2 Standard Sodium Hydroxide Solution, 5 N.

C-l.3 Nitric Acid, 1 *N*.

C-l.4 Glacial Acetic Acid

C-l.5 Acetic Acid in Methanol Solution, 5 percent (see B-2.5).

C-J.6 Preparation **of Pure MMA Reference Standard (see B-2.6).**

C-l.7 Ferric Chloride Solution (see B-2.7).

C-l.8 Phenolphthalein Indicator (see B-2.8).

C-1.9 Standard Solution of MMA (see B-2.9).

C-1.10 Blank Solution

Mix in a 100-ml volumetric flask IO-ml of acetic acid in methanol and 10 ml of ferric chloride solution and make up to the mark with methanol.

C-l.11 Comparison Solution

Pour 10 ml of the standard solution of MMA into a 100-ml volumetric flask, add 10 ml acetic acid in methanol, 50 ml methanol, 10 ml ferric chloride solution, mix well and fill up to the mark with methanol.

C-2 PROCEDURE

C-2.1 Take with a pipette 10 ml of the stock solution (see B-3.1) into a 100-ml volumetric flask and while keeping at 25 ± 5°C add 10'0 ml acetic acid in methanol solution, 50 ml methanol and 10'0 ml ferric chloride solution. Fill immediately with methanol to the mark and shake the solution thoroughly. This constitutes the test solution. Set the stop watch in motion. Pour the solution into a l-cm cell and measure the absorbance at 544 nm wavelength in a spectrophotometer. This measurement shall be done 30 seconds after the preparation of the solution is completed. The running of the absorbances shall be watched for the next 3 minutes at 30 seconds intervals. The obtained absorbance curve is extrapolated at the time 'zero'.

C-3 CALCULATION

C-3.1 $$K = 77'6 \times \frac{At \times Ms \times P}{As \times Mt \times 100}$$

At = absorbance of the test solution extrapolated at the time 0;

As = absorbance of the comparison solution;

Ms = mass, in g, of the reference standard (100 percent purity basis);

P = purity of the reference standard; and

Mt = mass, in g, of sample taken for the test.

C-3.2 Free aceto-acetic acid monomethyl amide (MMA), percent by mass = $\frac{K}{1{\cdot}94}$

NOTE — In the determination of free MMA, the absorption of the sample solution must not exceed 0·7. When higher concentrations occur, take 2·0 ml or 5·0 ml of the stock solution (see B-3.1). In the calculation, the dilution has to be considered accordingly.

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